Sharp vs Blunt Crack Hypotheses in the Strength of Glass: A Critical Study Using Indentation Flaws

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The fundamental question as to whether the tip structure of brittle cracks is atomically sharp or has a rounded contour is examined in relation to current descriptions of strengthcontrolling flaws. The distinction between the two opposing viewpoints lies in the controlling flaw dimensions in the strength formulation; crack length in the first and tip radius in the second. Definitive evidence on the issue is obtained from aging tests on soda-lime glass, using indentations as controlled flaws. An increase in the inert strength is observed with increased exposure of the newly created flaws to moist environments prior to stressing to failure. This strength increase saturates after approximately 1 day, depending on the environmental species. The trend mirrors that reported by Mould in an earlier aging study on abrasion flaws. However, whereas Mould concluded that the strengthening must be due to tip rounding, the present tests reveal that the indentation-induced cracks actually extend during the aging period. A fracture mechanics analysis shows that such extension relaxes residual crack-opening stresses associated with the central contact deformation zone. It is accordingly concluded that the cracks remain sharp throughout their postindentation evolution; the influence of extraneous conditions on the strength is manifested only through the driving forces on these cracks. Flaws which have been annealed (i.e., which have had their residual driving forces removed) show no such aging effects. The fracture mechanics analysis also shows that in the region of saturated aging, where the indentation cracks appear to stop growing, the fracture driving force is in the region of the zero-velocity threshold described in the macroscopic crack growth studies by Michalske. Contrary to previous interpretation, the indication is that the cracks do not blunt out in this region, for otherwise the strengthening would steepen rather than level out. Implications of this result concerning the inviolate nature of basic crack growth laws are discussed.

I. Introduction

T is now well accepted that the load-bearing capacity of glasses and other ceramics is limited by the almost inevitable presence of small flaws.1 The fundamental nature of these flaws has nevertheless remained a contentious issue, primarily because of the difficulty in observing their response to applied stresses in any direct way. Part of the difficulty is attributable to the small scale of the flaws (typically $\leq 100 \mu m$ in maximum dimension) and part to the fact that the location of the critical member in a large flaw population cannot usually be predetermined. Postfailure fractography of flaw origins, while useful as a means for quantifying the critical flaw sizes, tells us little about the micromechanics prior to instability. Consequently, modern-day theories of strength derive from simplistic models of the instability conditions, whereby the flaws are represented as stress-raising cavities with well-defined geometries. The following question remains: how well do such geometrical representations capture the essence of the fundamental failure processes? At stake here is our ability to under-

These flaw models have many variants, but all fall broadly into one of two schools of thought. The first school has it that the flaws are true microcracks with atomically sharp tips, so that the mechanics are governed by the measurable laws of crack growth. 2-5 In this description, strength is governed by an intrinsic "toughness" parameter, which measures the resistance of the material to crack extension, and an extrinsic crack *length* parameter, which quantifies the scale of the flaw. This picture constitutes the "sharp-crack" hypothesis. The second school differs from the first in that it rejects the notion of atomic sharpness, assuming instead that the tip regions are smoothly rounded, so that instability conditions are now governed by the laws of stress concentrations at notches. 6-9 This necessitates redefining the intrinsic strength parameter, but the major change is the replacement of length by tip radius as the critical flaw dimension (although the former dimension is not entirely eliminated from the description). This is the "blunt-crack" hypothesis. Unfortunately, the potential competition between crack lengthening and crack rounding in the mechanics to failure is not easily evaluated from strength data alone; on the face of it, both concepts appear to be capable of accounting for most observed trends in strength behavior. The facility to observe flaw response directly remains the key missing element in these evaluations.

In seeking a definitive experiment, we draw attention to two earlier fracture studies on soda-lime glass, both involving timerelated manifestations of attack by environmental water. The first of these studies concerns the progressively diminishing severity of flaws when exposed to moisture between initial formation and subsequent strength testing under inert conditions. In his classical paper on this so-called aging phenomenon, Mould⁶ showed that systematic strength increases of 30 to 60% (depending on detailed conditions of the experiment) could be realized with abrasion flaws. Mould considered three possible explanations: (i) the flaw undergoes some closure, (ii) residua! opening forces on the flaw are gradually relieved, and (iii) the flaw tip is blunted. Of these explanations, he favored the third, regarding the first two as "unlikely." (More recent investigations of aging properties have taken up the blunting theme more emphatically. 8.9) In the second study, by Michalske, 10 the velocity characteristics of cracks in large-scale test specimens were followed in the region of low sustained loads. It was observed in these experiments that the cracks tended to a zero-velocity threshold, in some historydependent manner, at a driving force approximately one third of that needed to cause unlimited extension in water-free environments. Michalske also interpreted his results in terms of tip blunting. Taken together, these two sets of results would seem to provide compelling, if circumstantial, evidence for the blunt-crack hypothesis, at least under conditions where water has access to the tip region.

In this paper we describe experiments which show that the above interpretations in terms of true blunting of crack tips are, at the very least, highly questionable. The approach adopted here, foreshadowed to a certain extent in recent studies by some of our co-workers¹¹⁻¹⁵ and by others, ¹⁶⁻¹⁸ is to introduce a single, dominant flaw into a prospective strength test specimen by indentation. The unique advantage of indentation flaws is their amenability to direct observation at all evolutionary stages, from initial formation to ultimate failure instability. Notwithstanding their "artificial" character, there is now considerable evidence to demonstrate that indentation flaws do indeed simulate the essential qualities of natu-

stand, and thence ultimately to control, the strength properties of brittle materials.

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rally occurring surface flaws in glass: flaws introduced by scratching (e.g., as in glass cutting), ¹⁹⁻²² by spurious particle impact, ²³⁻²⁵ and by finishing processes (abrasion, grinding, and machining)26-29 are just a few pertinent examples. Moreover, it has been shown that indentation flaws satisfy the laws of macroscopic crack growth over a wide size range, from millimeter- to micrometerscale,30 thus providing some justification for inferring potential flaw response from large-scale crack data. Our focus in this work will be on the way such flaws age on exposure to aqueous environments, using Mould's study as a comparative base for drawing general conclusions. Thus, we shall be led to assert that the strength increases associated with aging are unequivocally explainable in terms of residual stress relief and not of blunting. Although the actual strength measurements in our experiments are made under ostensibly inert testing conditions, our deliberations will be seen to bear strongly on fatigue properties, particularly in relation to the threshold region of crack growth described by Michalske.

It should be emphasized at the outset that the issues we seek to clarify here are not just exercises in semantics. It is only by establishing the underlying mechanisms of flaw response that we can hope to develop scientifically based criteria for predicting failure, especially under conditions which extend beyond the range of laboratory test data. An attractive corollary to our conclusions in favor of the sharp-crack concept is the inviolate nature of the critical tip region which controls the flaw evolution to failure. Thus in our picture the fundamental laws of crack growth remain uniquely expressible in terms of some characteristic driving force parameter (such as the stress intensity factor); it is only the driving forces themselves (mechanical, chemical, thermal, or otherwise) which are modified by extraneous conditions.

II. Sharp and Blunt Crack Hypotheses

In this section the opposing viewpoints of crack geometry as they relate to the question of strength are briefly surveyed. Insofar as our concern is with strength properties under inert testing conditions, we shall formulate our criteria for failure instability in terms of equilibrium fracture requirements, ³¹ although not so as to exclude the possibility of subcritical rate effects in the crack history prior to strength testing. The treatment is given in two parts, the first dealing with the two hypotheses in their elemental form and the second with the modifying influences of extraneous driving forces.

(1) Basic Models of Flaw Mechanics

Here we describe models of strength based on the sharp- and blunt-crack concepts, free of any complicating factors, such that the only driving force for failure comes from an applied tensile stress field. Figure 1 identifies the important variables for the two models: applied stress, σ_a , and crack length, c, are common to both; crack tip radius, ρ , is specific to the second model.

(A) Sharp-Crack Model: For the sharp-crack model depicted in Fig. 1(a) one may express the driving force for fracture in terms of an applied stress intensity factor

$$K_a = \psi \sigma_a c^{1/2} \tag{1}$$

where ψ is a dimensionless geometrical factor: e.g., for an ideal line crack embedded in an infinite medium $\psi = \pi^{1/2}$, ³¹ whereas for (Vickers) indentation cracks in glass ψ has a value close to unity. ¹³ In the absence of any subcritical rate-dependent effects, the crack remains stationary until the applied stress reaches some critical level, $\sigma_a = \sigma_f$ say, at which $K_a = K_c$, the material "toughness." At this point failure is spontaneous:

$$\sigma_f = K_c/\psi c^{1/2} \tag{2}$$

(B) Blunt-Crack Model: In the blunt-crack model of Fig. 1(b) the laws of stress concentrations at the tips of rounded notches, as initially laid down by Inglis,³² are assumed to apply. The concentrated stress for an ideal line crack is

$$\sigma_t = 2\sigma_a(c/\rho)^{1/2} \tag{3}$$

Failure at $\sigma_a = \sigma_f$ is now said to occur when σ_t reaches the theoretical cohesive strength, σ_c , of the material:

$$\sigma_f = \sigma_c \rho^{1/2} / 2c^{1/2} \tag{4}$$

This failure is again spontaneous, without any precursor change in the system dimensions.

The key distinction between these two models lies in the assertion as to which of the variables in Eqs. (2) and (4) actually control the strength behavior. Given that the material itself has invariant intrinsic properties, i.e., K_c or σ_c unchanged by experimental conditions (and even this assumption may require some qualification—see Section II(2A), below), the sharp-crack proponents seek to explain strength variations in terms of c; their blunt-crack counterparts regard c as a passive variable and explain the same variations in terms of ρ .

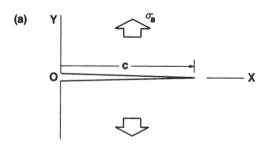
It is interesting to estimate, as Doremus has done, ⁸ the critical crack-tip radius, ρ_c , consistent with the blunt-crack view of failure. The argument goes that, whereas Eq. (2) is a *necessary* condition for failure, it is only Eq. (4) which is *sufficient*. Accordingly, the requirement that the strengths in Eqs. (2) and (4) should be identically equal yields (using $\psi = \pi^{1/2}$ appropriate to line cracks)

$$\rho_c = (4/\pi) \left(K_c / \sigma_c \right)^2 \tag{5}$$

which is interpreted as the *minimum* radius that an unstable crack may sustain. Now from toughness measurements on large-scale crack specimens in soda-lime glass we have $K_c = 0.75$ MPa·m^{1/2}, ³³ and from theoretical bond-strength computations (summarized in Ref. 8) $\sigma_c = 20$ GPa approximately, giving $\rho_c \approx 1.8$ nm, or about 5 molecular spacings (Si-O-Si distance = 0.32 nm). Of course, at subcritical stress levels the estimated radius is correspondingly smaller, $\rho \approx 0.2$ nm at $K = K_c/3$ (i.e., in the region of Michalske's zero-velocity threshold for tests in water).

In the context of aging phenomena, it would seem that the sharp-crack hypothesis as formulated in Eq. (2), based as it is on the precept of spontaneous instability of a constant geometry flaw,

[†]It should be pointed out that these values, insofar as they can ever be taken as physically realistic measures of crack-tip geometry, are likely to be overestimates: calculations based on an alternative representation of rounded tips, that of an initially closed slit opening under load into a parabolic contour, give values about one tenth of those above5; moreover, all such evaluations ignore the presence of attractive cohesive forces at the newly formed interface, which will tend to reduce the effective radius still further.³⁴



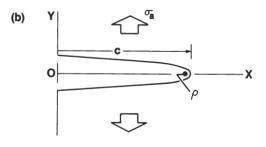


Fig. 1. Schematic showing essential variables for describing (a) sharp and (b) blunt cracks.

is unable to account for any possible increase in strength. The blunt-crack hypothesis, on the other hand, is not encumbered by a unique relationship between strength and crack length in Eq. (4); a strength increase can be explained in terms of an increase in tip radius above the minimum value, ρ_c .

(2) Models Incorporating Extraneous Driving Forces

We have indicated that the basic sharp-crack picture of flaw response does not possess the necessary "flexibility" to allow for the kind of strength variations observed in aging experiments. However, this flexibility may obtain if the flaws are subjected to extraneous driving forces, over and above that associated with the applied tensile loading, during the failure test. Here we consider two possibilities: (i) an interfacial closure force and (ii) a residual mouth-opening force. A strengthening trend could thus be brought about by either systematically increasing the first of these forces or decreasing the second. Actually, these modifications to the flaw description are of the same kind as those rejected by Mould in his original discussion of aging; 6 we shall be arguing later that this rejection was based on inadequate information concerning the flaw response.

(A) Interfacial Closure Forces: Consider a crack of characteristic length c subjected to closure tractions $-\sigma_i(x)$ across its interface, where x is a coordinate in the direction of crack expansion (Fig. 1). Then we may define an appropriate stress intensity factor in the manner of Barenblatt's formalism³⁴

$$K_i = -\int_0^c G(c, x)\sigma_i(x) \ dx \tag{6}$$

where G(c, x) is a Green's function.[‡] In a failure test this driving force superposes onto that associated with the applied loading (Eq. (1)) to give

$$K = K_a + K_i = \psi \sigma_a c^{1/2} + K_i = K_c \tag{7}$$

at equilibrium. Note that Eq. (7) may be reduced to the same form as the strength relations in Section II(1A) by substituting either $c' = c(1 + K_i/K_a)^2$ or $K'_c = K_c - K_i$, so that

$$\sigma_a = K_c/\psi c'^{1/2} = K_c'/\psi c^{1/2} \tag{8}$$

Since $K_i < 0$ (see Eq. (6)), these substitutions may be regarded as equivalent crack length reductions or material toughness increases, respectively. Although an explicit strength relation cannot be determined from this formulation without detailed knowledge of the functional dependence $K_i(c)$, it is clear that the basis exists for accommodating a progressive increase in the failure stress.

From a physical standpoint, the notion of an equivalent crack length reduction has a ready interpretation in terms of flaw healing, as originally suggested by Levengood. ²⁰ Healing could result from wall-wall cohesive interactions between pristine fracture surfaces or from wall-product adhesion at a chemically contaminated interface. There is experimental evidence confirming the occurrence of healing in glasses^{35,36} and other brittle materials. ^{4,37} An increase in material toughness may be realized by structural rearrangements in some "process zone," whereby an otherwise undisturbed flaw tip is "shielded" from the remotely applied loading. ^{5,38}

(B) Residual Mouth-Opening Forces: Now consider a crack subjected to a residual opening force at its mouth. Holland and Turner¹⁹ demonstrated the existence of such residual forces at fresh diamond scratches in glass by observing the tips of the attendant cracks in polarized light. They suggested that debris from the fracturing process was responsible for holding the cracks open. Recent studies of indentation flaw systems^{11,12,39} show that the source of residual opening is more generally associated with the irreversible deformation processes which accommodate the nearcontact displacement fields. An important observation in the fracture mechanics description of such systems is the tendency for the cracks to evolve predominantly during the unloading half-cycle¹¹;

this evolution is stable, so the cracks approach their immediate postindentation configurations in a state of true equilibrium. Hence, residual driving forces are a critical element in the underlying processes responsible for generating the flaws in the first place.

Basically, there are two types of cracks which identify with inelastic contacts. 40 The first is the "radial-median" crack, normal to the contact surface and coincident with the load axis. It is this crack type which is directly responsible for failures in subsequent strength tests and is accordingly of primary concern here. A detailed analysis of the fracture mechanics for *point* contact loading, such that the cracks ultimately assume a pennylike geometry (i.e., with semicircular fronts centered about the deformation zone), provides a stress intensity factor associated with the residual driving force 39

$$K_r = \chi P/c^{3/2} \tag{9}$$

where P is the contact force at maximum penetration, c is the radial crack dimension, and χ is a dimensionless factor which reflects the degree of elastic-plastic mismatch between deformation zone and surrounding elastic matrix. An analogous expression is determined for line contacts. The second crack type is the "lateral" crack, which extends from near the base of the deformation zone onto a plane closely parallel to the specimen surface. Although it does not lead directly to subsequent failures, the lateral crack, as we shall see, can become an important factor in the strength properties.

To formulate a theory for the subsequent strength of specimens containing dominant contact-induced radial-type flaws, we superpose Eqs. (1) and (9) to obtain the stress intensity factor¹²

$$K = \psi \sigma_a c^{1/2} + \chi P / c^{3/2} = K_c \tag{10}$$

for equilibrium configurations. The residual stress term in this expression, by virtue of its inverse dependence on c, exerts a stabilizing influence on the crack growth. This stabilization may be demonstrated formally by rearranging Eq. (10) to obtain the function $\sigma_a(c)$ and setting $d\sigma_a/dc = 0$; this determines a maximum at

$$\sigma_m = 3K_c/4\psi c_m^{1/2} \tag{11a}$$

$$c_m = (4\chi P/K_c)^{2/3} (11b)$$

The failure condition now depends on whether the flaw size c immediately prior to application of the applied tensile loading is less than or greater than c_m . If $c \le c_m$, the flaw grows stably with increasing σ_a until $\sigma_a = \sigma_m = \sigma_f$; Eqs. (11a) and (11b) then combine to give the instability stress

$$\sigma_f = 3K_c^{4/3}/4^{4/3}\psi\chi^{1/3}P^{1/3} \tag{12a}$$

Conversely, if $c>c_m$, failure is spontaneous at $\sigma_a=\sigma_a(c)=\sigma_f$, i.e.,

$$\sigma_f = (K_c/\psi c^{1/2})(1 - \chi P/K_c c^{3/2})$$
 (12b)

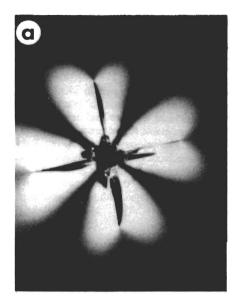
Note that the strength in Eq. (12a) is independent of starting flaw size c, but falls off thereafter in Eq. (12b) as c is allowed to exceed the critical depth c_m .

An increasing trend in strengths would now follow from Eq. (12) if the residual stress intensity, as quantified by the parameter χ , were somehow to be reduced below its immediate postindentation level. Reductions in χ can in fact be realized by specific treatments which effectively remove the source of the residual stresses, i.e., the deformation zone: progressive surface polishing, ⁴¹⁻⁴³ annealing, ^{12,13,16} and acid etching ¹⁷ are examples of such treatments. It is not immediately clear how any reductions of this kind might occur under typical aging conditions, however, and therein may be said to lie one of the major causes for dismissal of residual stress effects in the earlier strength analysis literature.

III. Aging Experiments

The preceding theoretical formalism provides us with the necessary background for analyzing the results of aging data on controlled flaws. At this point it is appropriate to elaborate on the main

[‡]For instance, for indentation cracks with pennylike geometry, $G(c,x) = \psi \alpha / c^{1/2} (c^2 - x^2)^{1/2}$. Note that if we put $-\sigma_i(x) = \sigma_a = \text{constant}$ into this integral, we obtain $K_i = \psi \sigma_a c^{1/2} = K_a$, the stress intensity factor for uniform tensile stress in Eq. (1).



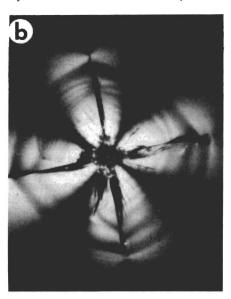


Fig. 2. Vickers indentations in soda-lime glass, P = 49 N, (a) immediately after contact and (b) after aging in moist environment for 1 h (courtesy D. B. Marshall).

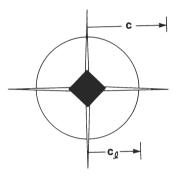


Fig. 3. Schematic indicating crack dimensions measured in indentation aging experiments, showing radial (primary) crack, c, and lateral (secondary) crack, c.

findings of Mould, whose work using abrasion flaws on soda-lime glass stands as an important precursor to our own study. Mould found the following:

- (i) The inert strength of freshly abraded surfaces increases rapidly on exposure to moist environments;
- (ii) This strength increase decelerates with time, with a tendency to plateau out after ≈ 1 day;
- (iii) The magnitude of the strength increase is typically 30% for point (grit-blast) flaws, somewhat higher for line flaws;
- (iv) Annealing of the abraded surfaces produces strengths about equal to the upper limit of aged flaws;
- (v) No further strength increase occurs on aging annealed flaws.

As mentioned in Section I, Mould was led to conclude that the strength increases observed were the result of crack-tip rounding, by "dissolution" in water environments and by "atomic rearrangements" at elevated temperatures. He pointed out that this rounding would need to involve just one or two molecular layers to account for the scale of the effect. The absence of a continued strength increase after annealing was attributed to a reduced wettability of the glass surfaces due to dehydration.

The experiments to be described below will take us to an altogether different conclusion as to the underlying mechanism of aging.

(1) Experimental Procedure

The controlled flaws in our experiments were introduced by using a standard Vickers diamond pyramid indenter. The speci-

mens were soda-lime glass bars approximately 125 by 15.5 by 5.5 mm, preannealed to remove any spurious surface stresses. For the aging tests proper, a single indentation was centrally placed in the upper surface of each bar, such that one of the two mutually orthogonal sets of radial cracks would be oriented normal to the tensile axis in the ensuing strength test. A few "control" specimens were prepared with *three* indentations spaced 5 mm apart along the longitudinal center line, for investigation of the failure instability conditions. ⁴⁴ A fixed load of P = 49 N was chosen for all tests, such that the radial cracks should be sufficiently well developed as to constitute the dominant flaw, yet not too large as to be a significant fraction (i.e., more than one tenth) of any specimen dimension.

The indented specimens were then subjected to a prescribed aging treatment. Distilled water was the principal aging environment, but silicone oil and a 1% HF/1% H₂SO₄ solution were also used, as comparative inert and corrosive extremes, respectively. In the case of the water and acid solutions, the specimens were totally immersed in holding baths immediately after indentation in air. For the oil (where evaporation is not a problem), a droplet was simply placed onto the predetermined contact site immediately before indentation. The cracks in some of the specimens in water and oil were monitored at selected intervals during the aging treatment (taking care to prevent the specimen surfaces from drying out at such times) with a polarizing microscope. ¹¹ This kind of observation, typified in Fig. 2, reveals a strong postindentation expansion of both the radial and lateral systems. ^{13,14,43} Accordingly, the respective crack dimensions, c and c_l , were recorded as depicted in Fig. 3.

In line with Mould's experimental course, a group of specimens was given a further annealing treatment before strength testing. These specimens, after a prolonged (>2 day) exposure to moist environment (to allow the crack system to achieve a "saturated" state—see Section III(2) below), were baked in air at 520°C for 24 h. 12.13 They were then subjected to additional aging in water, in the manner described above.

Finally, the specimens were broken in four-point flexure, using an inner span of 38 mm and outer span of 107 mm, with the indented face on the tensile side. Near-inert conditions for these tests were ensured by covering the indentation sites with silicone oil and keeping the failure times below 10 s.^{30} For the specimens aging in water or acid solution this meant, of course, that the indentation had first to be dried out (in our case, in a hot air stream), thus limiting the minimum time between indentation and flexure. Some of the annealed specimens were put through a preliminary flexure cycle in air so as to produce a minute yet perceptible amount of slow crack growth (typically 5 μ m, or <2% of the total crack length), to provide a reference base for investigating

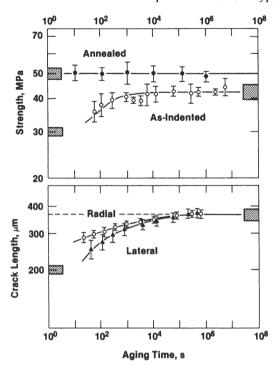


Fig. 4. Aging of Vickers-indented soda-lime glass in water, P = 49 N, showing strength and radial and lateral crack dimensions as functions of flaw lifetime. Data represent as-indented and postindentation-annealed specimens. Hatched regions denote limiting flaw configurations (see text).

potential blunting during the heat treatment. An examination of the broken bars confirmed the indentation sites as the source of all the failures in this study.

(2) Results

The results of the present study are summarized in Figs. 4 to 6. Each data point on these plots denotes the mean and standard deviation (computed in logarithmic coordinates) of measurements from at least five specimens. The hatched regions identify important limiting flaw configurations, representative of immediate postindentation, fully aged, and annealed states (designated hereafter by superscripts 0, ∞ , and A, respectively), for later analysis.

Consider first the strength trends for the aging tests in water (Fig. 4). A systematic increase with time is immediately apparent for the freshly indented specimens. This increase from the immediate, zero-age-time strength $\sigma_0^0 = 30.0 \pm 1.2$ MPa (evaluated from Fig. 5—see below) is initially rapid, but slows down thereafter until, after several hours, a saturation level $\sigma_0^{\pi} = 42.2 \pm 2.4$ MPa is approached. The strength of the well-aged specimens increases further on annealing, to $\sigma_0^A = 49.9 \pm 2.4$ MPa, but remains constant at this level with additional aging. Those annealed specimens subjected to a preliminary, crack repropagation load cycle gave strengths in the same range, 48.2 ± 3.5 MPa.

It will be noted that these results are identical in all qualitative respects with the major findings of Mould on abraded surfaces, enumerated earlier. The only differences appear in the *degree* of strengthening; our increases of \approx 40% for fully aged and \approx 60% for annealed specimens are a little higher than for Mould's grit-blast specimens. This strong parallel in behavior is not too surprising, considering our indication earlier (Section I) that indentation flaws simulate many of the essential characteristics of natural flaws. Now, however, we have the unique advantage of being able to watch what happens to our flaws during the strengthening process.

Before attempting to relate the aging trends to actual crack observations, it is instructive to compare the strength data for water with those for the other aging media. For the tests in oil (Fig. 5)

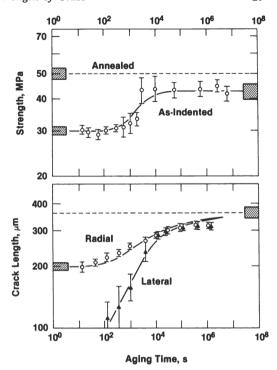


Fig. 5. Aging of Vickers-indented soda-lime glass in silicone oil, P=49 N, showing strength and radial and lateral crack dimensions as functions of flaw lifetime. Data represent as-indented specimens; levels for postindentation annealing state are taken from Fig. 4. Hatched regions denote limiting flaw configurations.

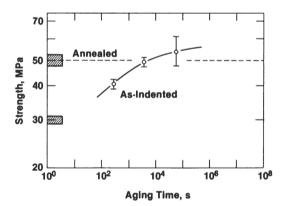


Fig. 6. Aging of Vickers-indented soda-lime glass in HF solution, P=49 N, showing strength as function of flaw lifetime. Data represent as-indented specimens; level for postindentation annealing state is taken from Fig. 4. Hatched regions denote limiting flaw configurations.

we see that the trend is much the same as before insofar as the saturation level is concerned, but that the time to reach this level is considerably greater. It is as if the aging curve has been simply displaced to the right along the horizontal axis. This suggests that the mechanism of strengthening is unchanged, so the oil does no more than regulate the accessibility of trace water to the crack-tip region. Indeed, it appears from Fig. 5 that this retardation of the active species is sufficiently pronounced as to inhibit any significant development of the flaw system for several tens of seconds, thereby allowing for convenient evaluation of the immediate post-indentation state.

For the tests in acid solution (Fig. 6) the rate of strength increase is, if anything, a little higher than for water. Moreover, in this instance the *degree* of strength increase is also higher, up around

(or even slightly in excess of, owing to some reduction in the crack depth^{17,18}) the level for annealed specimens. These acid-aged specimens showed strong preferential etching at the central deformation zone and along the radial crack traces.

Now let us examine the crack expansion data in Figs. 4 and 5 in the light of the strength trends just described. It is immediately clear that a strong correlation exists here. Broadly speaking, the strongest strength increases occur where the crack growth is fastest. For the annealed indentations this correlation is hardly dramatic—zero strength change, zero crack growth. For the newly formed indentations, on the other hand, we may identify three regions on the plots where the aging response undergoes distinctive changes in character:

- (i) An "incubation" region, during which the strength remains effectively at its immediate postindentation level. This region is seen to best advantage in the oil-environment data (Fig. 5). The radial cracks are correspondingly inhibited in the expansion from their initial, zero-aged configuration at $c^0 = 195 \pm 12 \,\mu\text{m}$, consistent with the notion of a lead time for penetration of water alluded to earlier. Special note may be made of the relatively late development of the lateral crack system which, as we recall from Section II(2B), has its origins in the subsurface deformation zone.
- (ii) A "rapid-aging" region, during which the bulk of the strength increase occurs. The bulk of the crack expansion is also apparent in this region. The environmental dependence of the expansion *rate* in the data is consistent with macroscopic crack velocity trends, ^{2,45-47} where water is identified as a principal interacting species. A feature of significance in the crack evolution, again seen most clearly in Fig. 5, is the tendency for the lateral system to overcome its initial sluggishness and ultimately outgrow its radial counterpart.
- (iii) A "plateau" region where, after extended aging, the strength appears to saturate. The radial cracks likewise appear to saturate in their growth, at $c^{\infty} = 362 \pm 25 \ \mu \text{m}$ (although the accuracy of our measurements is such that continued extension at a velocity $\leq 10^{-11} \text{ m} \cdot \text{s}^{-1}$ would go undetected).

Finally, it will be recalled from Section III(1) that some control specimens were prepared with three indentations. These specimens were broken in the immediate postindentation and fully aged states, i.e., in regions (i) and (iii) of the aging response, to gain some clue as to the crack stability conditions during the strength test itself. Since failure initiates from just one of the indentation sites, each specimen contains two "dummy" flaws which have been on the verge of instability. Inspection of such specimens showed substantial precursor radial crack extension for the newly formed indentations, by well over a factor of 2 in size, but no detectable extension at all for the fully aged indentations.

IV. Interpretation of Aging Results

Consider now how the aging results in Figs. 4 to 6 may be reconciled with the sharp- and blunt-crack hypotheses outlined in Section II. Our major finding is that the strength increases correlate with postindentation crack expansion. This would immediately appear to rule out two of the three possible explanations for such increases offered earlier: neither the blunt-crack concept (Section II(1B)), with its assertion that the crack-tip radius ρ constitutes the primary strength-controlling variable, nor the notion of a crack with superposed closure forces (Section II(2A)), with its connotation of a retraction in length (real or effective), is compatible with such an expansion. On the other hand, an extensive stage of postindentation growth is perfectly consistent with the residual driving force model (Section II(2B))^{13,48}; indeed, by combining Eq. (9) with a crack velocity relation appropriate to the aging environment, it is possible to obtain quantitative estimates of this growth. 49 We are left with the question: how can an increase in crack size lead to an increase in strength?

The answer to this question lies in the behavior of the component of the crack system which is *not* directly responsible for failure, i.e., the *lateral* component. The strength formulation of Eq. (12) contains explicit reference only to the *radial* crack dimension, c: there is provision in this formulation for σ_f to diminish with c

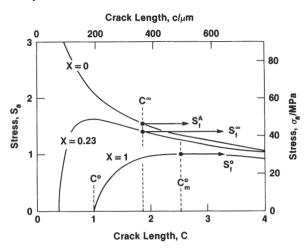


Fig. 7. Plots of applied stress vs radial crack length for equilibrium indentation flaws, $P=49\,\mathrm{N}$, in soda-lime glass. Curves are solutions of Eq. (10) for three limiting flaw states, immediate postindentation (lower), fully aged (intermediate), and annealed (upper). Arrows designate appropriate failure configurations. Failure configuration for immediate postindentation state establishes reference base for normalization of variables.

(Eq. 12b), or, if $c \le c_m$, even remain independent of c (Eq. 12a), but not increase with c. However, a strength increase can obtain if the residual stress parameter χ in Eq. (12) undergoes some progressive diminution in the postindentation period. Roach and Cooper, 16 in a key, quantitative study of stress birefringence around indentation sites, did in fact find a strong inverse relationship between strength and optical retardation over a wide range of aging conditions. The growth of lateral cracks is an effective means of reducing the residual stress intensity, 13,19,21,43,50 by relaxing the elastic-plastic mismatch displacements at the deformation zone boundary. Recall that the strength data in Figs. 4 and 5 exhibit their strongest increasing trends in the region where the initially sluggish lateral cracks begin to overtake their radial counterparts. Of course, the facility to observe subtle crack evolution characteristics of this kind was not available to Mould in his study of abrasion flaws, so it is hardly surprising that he was led to reject the residual stress hypothesis.

Having thus identified c and χ as our independent variables for the postindentation condition, it is of interest to investigate the fracture mechanics formulation of Section II(2B) in terms of the data for soda-lime glass in Section III. For this purpose, it is convenient to adopt the immediate postindentation state as a base for comparing the fully aged and annealed states. Accordingly, we may usefully define the reduced variables $C = c/c^{\circ}$ and $X = \chi/\chi^{\circ}$; the corresponding reduced stress $S_a = \sigma_a/\sigma_f^{\circ}$ may then be determined from Eq. (10) as a unique function of C for any given value of X:

- (i) Immediate Postindentation State. Since the newly created indentation flaw begins its life in equilibrium, we may insert $\sigma_a = 0$ into Eq. (10) to obtain $c^0 = (\chi^0 P/K_c)^{2/3} = 195 \pm 12 \ \mu m$, and thereby establish the reference crack length $C^0 = 1$. This in turn establishes the reference residual stress parameter $X^0 = 1$. On applying the tensile loading in the strength test the crack grows stably, to a critical size $C_m^0 = 4^{2/3}$ according to Eq. (11b). This scale of precursor growth is consistent with that actually observed in the dummy indentation tests. The strength relation appropriate to this kind of crack response is Eq. (12a), from which we have $\sigma_f^0 = 3K_c^{4/3}/4^{4/3}\psi\chi^{0.1/3}P^{1/3} = 30.0 \pm 1.2$ MPa, corresponding to $S_f^0 = 1$. A graphical interpretation of the failure path is obtainable from the lower curve in Fig. 7, representing Eq. (10) at X = 1.
- (ii) Fully Aged State. On prolonged exposure to moist environment the flaw undergoes subcritical postindentation radial growth to $c^{\infty}=362\pm25~\mu\text{m}$, i.e., $C^{\infty}=1.86\pm0.17$. In this case the dummy indentation strength tests show no evidence of any

further, stable development. Thus the appropriate strength relation is that for spontaneous failure from the stationary flaw (Eq. (12b)). In conjunction with our definition of σ_0^0 in (i) above, along with the measured plateau strength $\sigma_f^\infty=42.2\pm2.4$ MPa, Eq. (12b) reduces to $S_f^\infty=(4^{4/3}/3C^{\infty\,1/2})\,(1-X^\infty/C^{\infty\,3/2})=1.41\pm0.10,$ from which $X^\infty=0.23\pm0.17$ is evaluated (i.e., the residual driving force in reduced to about a quarter of its optimum, immediate postcontact level). We may note that Eq. (11b), in conjunction with c^0 above, likewise reduces to $C_m^\infty=(4X^\infty)^{2/3}=0.95\pm0.47,$ which lies well within the requirement $C^\infty>C_m^\infty$ for validity of Eq. (12b). The failure condition for such fully aged flaws is represented by the intermediate curve in Fig. 7.

(iii) Annealed State. The annealing treatment is carried out on aged indentations, so the starting flaw size for the strength tests is the same as in (ii), i.e., $C^{\infty} = 1.86 \pm 0.17$. The residual stress parameter for this state is, of course, $X^A = 0$. Equation (12b) now takes on an even simpler normalized form, $S_f^A = 4^{4/3}/3C^{\infty 1/2}$, from which we calculate $S_f^A = 1.55 \pm 0.07$ as our relative strength level. The experimentally determined strength $\sigma_f^A = 49.9 \pm 2.4$ MPa gives the comparable value $S_f^A = 1.66 \pm 0.10$, and thereby affords a useful consistency check for the fracture mechanics formulation. This state is represented by the upper curve in Fig. 7.

With these flaw states so evaluated, we may take a closer look at the extent of the residual stress relaxation that attends the aging process in soda-lime glass. The mechanics of flaws in the post-indentation region are governed by Eq. (9), which can be rewritten in the form

$$K_r = K_c X / C^{3/2} (13)$$

Hence in the freshly created state, $C^0 = 1$ and $X^0 = 1$, the residual stress intensity factor is at the critical level for extension, $K_r^0 = K_c$, but falls off thereafter as the radial and lateral crack components expand, i.e., as C increases and X decreases. On reaching the fully aged state, $C^{\infty} = 1.86 \pm 0.17$ and X^{∞} 0.23 ± 0.05 , the driving force saturates out at $K_r^{\infty} = (0.09 \pm 0.05)$ $(0.07)K_c$. This saturation in the flaw evolution is consistent with Michalske's zero-velocity threshold in large-scale crack specimens, 10 although his cutoff stress intensity factor of $\approx K_c/3$, for nominally the same material-environment system as studied here, is somewhat higher than our estimate. In view of Michalske's own observation that the threshold conditions can be strongly history dependent, and allowing that the accuracy of the indentation equations is contingent on the validity of certain implicit assumptions (e.g., that the flaw geometry parameter ψ in Eq. (12) remains constant throughout the aging process), such a discrepancy hardly detracts from the striking parallel in qualitative behavior; the indentation flaw system conveniently takes us, through the natural course of its postindentation development, progressively down the v-K curve to the very threshold region which ultimately limits the aging.

This interpretation of the long-term aging limit raises more questions concerning the blunt-crack concept, for it will be recalled from Section I that Michalske considered his threshold phenomenon to be a manifestation of tip rounding. 10 Now if the contention that blunting is the underlying cause of aging⁶⁻⁹ were indeed to be correct, Michalske's conclusion would lead us to expect the strength increases to be optimal when the flaw has ceased to grow, not while it is still expanding, i.e., precisely the reverse of what is observed. Quantitative estimates of these optimal strength increases serve only to highlight the inconsistency here. We recall from Section $\Pi(1B)$ that the tip radius is computed to be of submolecular dimensions at the zero-velocity threshold; according to Eq. (4), removal of one additional molecular layer from the crack walls would thereby raise the strength by at least a factor of $\sqrt{2}$. No strengthening of this magnitude is evident in the saturation regions of Figs. 4 and 5, even for soak periods of up to 3 months in the aging medium. In the context of the residual-stress description, it is to be remembered that the instability condition for fully aged flaws falls in the class of spontaneous failure; i.e., the critical configuration on the appropriate $\sigma_a(c)$ curve in Fig. 7 lies to the right of the functional maximum, so this predicted sensitivity to the

initial flaw geometry should still be apparent in the strength data. It has to be concluded that crack-tip rounding has nothing whatever to do with the aging mechanism.

Moreover, there is the observation that specimens which have been annealed do not show strength increases on subsequent exposures to aging environments. Mould suggested that this null effect was due to a reduced wettability of the glass as a result of thermally induced dehydration. However, the results for the annealed indentation flaws lie in the same range regardless of whether or not a preliminary flexure cycle was administered prior to the actual failure test (Section III(1)); the fact that some slow crack growth could be produced during such a cycle demonstrates that atmospheric moisture has no difficulty in finding its way to the tip region. It is apparent that the important effect of the annealing here is simply to relieve all residual stresses about the flaws and thereby to remove the essential driving force for aging. In further support of our argument, we may point out that the same null aging effect is evident in freshly created cone cracks 15; cone cracks are similar to our annealed radial cracks in that they are free of residual stresses (the original contact field being perfectly elastic⁴⁰), vet without ever having been exposed to conditions which might have caused dehydration.

This leaves us to account for the strength trends observed in Fig. 6 for an acidic aging solution. The HF component of this solution is, of course, one of the strongest known glass corroding agents, so the crack blunting hypothesis, if it is to retain any credence at all, must surely manifest itself here. Yet the strength levels in Fig. 6 barely rise above that for the anneal treatment, suggesting that the acid does little more than etch away the central source of residual stress. It is not as though the solution is precluded from entering the cracks; we have already remarked on the preferential dissolution at the surface radial traces (Section III(2)). However, it would seem that the cracks somehow manage to remain sharp along the deeper portions of their fronts. Roach and Cooper, 17 in a more extensive study of the effects of etching on indentation flaws, arrived at much the same conclusion. They delineated two stages of increasing strength: in the first, effectively duplicated by the results in Fig. 6, the increase is relatively slow, and can be accounted for fully in terms of residual stress relief, plus some reduction in the crack depth; in the second, marked by the point at which the etch depth ultimately exceeds the original crack depth, the strength accelerates rapidly, in accordance with expectations from the theory of rounded notches. It is somewhat ironical that this apparent immunity of the subsurface crack front to acid attack was foreshadowed in study of natural flaws by one of the strongest proponents of tip dissolution in other, less corrosive solutions.

Thus our interpretation of aging properties would seem to imply a certain immutability in the nature of truly brittle cracks, consistent with the notion of atomic sharpness. Once formed, flaws with such characteristics are not easily blunted out, except of course by total physical removal. On the other hand, the forces which drive the flaws in their evolution to failure are most susceptible to extraneous mechanical, chemical, or thermal influences and are therefore likely to hold the key to any systematic strength variations involving such extraneous variables as aging time.

Finally in this section, some comments on the generality of the present indentation results are in order:

(i) Extension to Naturally Occurring Flaws. The point was made earlier (Section I) that indentations simulate many of the essential qualities of natural flaws, especially those produced in surface finishing operations and under service conditions of erosive wear. It is clear that the vital element necessary for a strength increase is the persistence of a component of the local stress fields originally responsible for creating such flaws. A clue as to the qualitative similarity between indentation and other surface flaw types is to be found in the tendency for the latter to be accompanied by chipping, 40 associated with the upward growth of lateral segments in particularly severe flaw generation events. Indeed, the success of most current theories of erosion and machining removal rates rests with a formal fracture mechanics analysis of this kind of limiting lateral crack development. 51 Of course, any chipping ap-

parent in the immediate postgeneration state (likely to be accentuated where overlap between adjacent flaw sites is substantial) must reduce the capacity for further stress relaxation in the ensuing flaw evolution, which could explain why Mould's grit-blast specimens showed a lower degree of aging than did our indentation specimens.

(ii) Point vs Line Flaws. Our focus thus far has been on point-contact loading. The course followed in Section II(2B) can be used to obtain an analogous strength formulation for line-contact loading, replacing $c^{-3/2}$ by $c^{-1/2}$ as the crack-size dependence of K_r in Eq. (9). ^{22,52} This longer range influence of the residual stress term translates into an increased potential for strength increase due to subsequent relaxation of the χ parameter. ⁵² Mould's observation of a significantly stronger effect for lineabrasion flaws relative to grit-blast flaws (Section III) is in accord with this predicted trend.

(iii) Effect of Flaw Size. The tests reported in this work were all run at a fixed indentation load of 49 N. Comprehensive studies of the strength properties of specimens indented at different loads indicate that flaw size is not an important factor insofar as the validity of macroscopic crack growth laws is concerned, at least when c falls in the micro- to millimeter range. 30 Some additional aging tests run by one of our colleagues (Ref. 53) at several loads between 2 and 100 N do indeed show similar strength trends to those found here. Exceptions to this implied universality in behavior are evident at the extremes of the crack size range cited above: with large flaws the tendency to chipping restricts the aging process, in the manner already discussed in (i); with small flaws we begin to enter the region of "subthreshold" behavior, where the response is governed more by the mechanics of crack initiation than propagation.54

(iv) Other Glasses and Ceramics. We have looked only at soda-lime glass here. Do our conclusions extend to other glasses and ceramics? In addressing this question it may be noted that soda-lime glass has so-called "normal" deformation properties, in that the indentation displacements are accommodated by a volumeconserving shear process. In silicate glasses without a significant network modifier content, however, the deformation takes on "anomalous" characteristics, with accommodation by a structural densification mode. This latter mode is much less effective in generating a residual stress field.55 Some workers have accordingly been led to suggest that the model of stress relaxation by postindentation lateral crack growth may not be appropriate for explaining aging effects in high silica glasses. However, a recent series of aging tests on fused-silica specimens with a variety of flaw types reveals exactly the same qualitative trends in the strength responses as for soda-lime glass. 15 Since the existence of residual contact fields is a general material phenomenon, 39 it must be expected that aging effects will also be manifest in other ceramics which are susceptible to slow crack growth, although it is conceivable that in most cases the approach to a saturated configuration may occur so rapidly (owing to steepness in the v-K curve) as to pass unnoticed.

V. Implications Concerning Fatigue Limits in Crack Velocity Characteristics

Our conclusions above concerning the inapplicability of the blunt-crack concept relate specifically to equilibruim fracture configurations, for the strengths in the aging experiments are measured under essentially inert environmental conditions. The question may be asked as the what extent these conclusions extend to fatigue fracture configurations, where slow crack growth effects become manifest in the strength characteristics, i.e., where the failure mechanics are determined by the crack response at subcritical K levels. In particular, how do we reconcile our saturation in long-term aging response with Michalske's interpretation of his crack velocity threshold?¹⁰

Specifically, we have intimated that the absence of substantial strength increases after the indentation cracks have ceased to grow (Figs. 4 and 5) weighs against an explanation in terms of tip rounding. Some might argue that rounding does indeed occur in the

threshold K region, but that on raising K once more the cracks rapidly "resharpen" (presumably because of some residual water in the vicinity of the tips), so that details of the initial flaw state would not reflect sensitively in an (ostensibly) inert strength test. (The resharpening distance would, after all, be small, i.e., approximately one crack-tip radius, or molecular dimensions.) This is tantamount to conceding that crack-tip radius has no place in the general mechanical description of failure instability, consistent with our contentions in the previous section. More detailed considerations of the potential competition between crack sharpening and crack extension in the moisture-assisted strength degradation of glass 14,56 lend quantitative weight to these conclusions. The only aspect of the strength behavior which is critically dependent on the mechanics in the ν -K threshold region is the issue of the fatigue limit itself.

The questions that our work raise concerning the crack-blunting argument should not be seen as detracting from the significance of Michalske's threshold experiments, for the existence of a fatigue limit does have important repercussions in long-lifetime engineering design. It is to be emphasized that the results in Section III do not allow us to make any definitive statement as to the exact nature of the crack velocity cutoff. However, there is the strong underlying suggestion that the true explanation is to be sought in the reduction of some component of the overall fracture driving force. Such a reduction could be brought about by the progressive build up of chemically active species within the constrained crack interface at the lower velocities. For instance, wall-wall attractions could generate in the manner mentioned earlier in relation to Eq. (6), thus producing a closure force which ultimately, in the saturation region, is sufficient to negate the applied loading. The observation that cracks can undergo some healing (even in the presence of contaminating environments^{35,36}) lends credibility to this possibility. Alternatively, the presence of a corrosion product at the interface could have a retarding effect on the passage of water to the crack tip, leading to a similar response at the low end of the v-K curve. The apparent immunity of crack tips to etch attack in HF solution (Section IV) fits in with this picture. In the light of our estimates of crack-tip radii in Section II(1B), particularly in the region of the zero-velocity threshold, it is envisaged that the thickness of the active chemical layer could be as small as one or two molecular dimensions, i.e., within the range of typical adsorption layers.

Two other points cited by Michalske in support of the blunting viewpoint¹⁰ warrant attention here. The first of these concerns the observation of hysteresis in the crack velocity response. On cycling the driving force down and back up through the threshold configuration there is a measurable delay time before the crack system regains its position on the original v-K curve. The second point concerns the appearance of "tear lines" on the fracture surfaces marking the position of the arrested crack front after being subjected to such a hysteretic cycle. It is argued that these characteristics are symptomatic of crack reinitiation from a rounded notch. However, evidence of this kind has to be recognized as circumstantial. The interfacial chemical picture offered above would seem to allow for an equally plausible explanation of the phenomenology. A threshold incubation time results if the processes actually responsible for the buildup of the chemical layer have any rate dependence (as would be the case if, for example, the diffusion of charged species were to be involved). The generation of tear lines simply requires that these same processes should develop some kind of spatial instability in the crack system (in much the same way as is implicit in the reinitiation argument), so that the repropagation might occur at preferential sites along the crack front.

The indications are therefore very strong that atomically sharp cracks retain their structural integrity even under the most adverse fatigue conditions. Processes usually associated with tip rounding, such as corrosive dissolution, are confined to the region *behind* the tip. Accordingly, such processes play no direct role in the fatigue properties, except in determining a lower limit to the long-term strength. This is not to say that active species which promote slow crack growth, notably water, are denied access to the critical crack-

tip region. Rather, it is to assert that, by virtue of the "channelling" effect of the ever-narrowing crack walls,57 the chemical interactions become more specific to those bonds which exist in a state of high mechanical strain, thereby enhancing the brittle mechanism of sequential bond scission. In this view the fundamental relations between crack velocity v and driving force K are uniquely determined by the nature of the interactions in the near region of the crack-tip bonds themselves. The zero-velocity threshold and other such apparent departures from these relations simply reflect the influence of secondary driving or restraining elements on mechanical and chemical conditions at the crack tip. The dichotomy between mechanisms and mechanics is appropriate here: the former has the quality of invariance; it is only the latter which is subject to the influence of extraneous forces on the crack system.

VI. Conclusions

- (i) Our experiments have been run with controlled indentation flaws, so the progress of cracks could be followed directly at all stages of their evolution. These experiments demonstrate that residual driving forces are an important factor in the postindentation response, but that these residual stresses diminish as the cracks continue to grow. Other, natural flaw systems, particularly those with a contact-related history, are expected to behave in essentially
- (ii) The aging data on indentation flaws mirror the trends observed by Mould for abraded surfaces in all respects. In particular, a time-dependent strength increase is observed only for freshly created flaws; annealed flaws show no aging effects. This strength increase is shown unequivocally to be associated with relaxation of the residual driving force due to crack growth; when the cracks slow down, so does the aging. It is accordingly concluded that the cracks must remain sharp throughout the aging treatment.
- (iii) In the saturation region for fully aged flaws the cracks attain the zero-velocity threshold configuration on Michalske's v-K curve. The absence of further strengthening in this region implies that the cracks are not blunting. This in turn implies that the velocity cutoff is associated with some reduction of the net crack driving force. There is the suggestion that the source of such a reduction lies with interfacial interactions behind, rather than at, the crack tip.
- (iv) It is concluded that atomically sharp cracks have the quality of immutability, so that the basic laws of crack growth are inviolate. The explanations for apparent deviations from these laws are to be sought in the fracture mechanics accounting, not in the nature of the critical chemical interaction. This would seem to provide a strong motivation for continuing crack growth studies on idealized, macroscopic test specimens and for developing more sophisticated atomic models of crack-tip structures.

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